

## Tocopherol/Tocotrienol - WPI Results

L22 ANSWER 13 OF 222 WPIDS COPYRIGHT 1997 DERWENT INFORMATION LTD  
AN 94-256615 [32] WPIDS  
DNC C94-121037

TI Sepn. of tocopherol and sterol from, e.g. sunflower oil distillates  
- including a step involving esterification of the sterols with  
fatty acids present in the distillates.

DC D23 E13

IN FIZET, C

PA (HOFF) HOFFMANN LA ROCHE & CO AG F; (HOFF) HOFFMANN LA ROCHE INC  
CYC 13

PI EP 610742 A1 940817 (9432)\* DE 11 pp  
R: AT BE CH DE DK ES FR GB IT LI NL

JP 07002827 A 950106 (9511) 9 pp  
US 5487817 A 960130 (9611) 7 pp

ADT EP 610742 A1 EP 94-101257 940128; JP 07002827 A JP 94-34234 940208;  
US 5487817 A US 94-185571 940121

PRAI CH 93-467 930211

AB EP 610742 A UPAB: 941010

Sepn. of tocopherols and sterols from fat residues from vapour sepn.  
for diodorisation comprises (a) esterification of the sterols in the  
fats with fatty acids which are also present; (b) distillation of  
the resulting mixt. to recover residual fatty acids and to recover  
tocopherols, leaving the sterol esters in the distillation residues;  
and (c) isolation of the tocopherols from the distillate and  
isolation of the sterols, after cleavage of the esters, from the  
distillation residues.

USE/ADVANTAGE - The tocopherol prods. have vitamin E activity.

The process allows recovery of the tocopherols and sterols in  
separate stages, and are easier and cheaper than previous processes.

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Tocopherol/Tocotrienol - WPI Results

L22 ANSWER 19 OF 222 WPIDS COPYRIGHT 1997 DERWENT INFORMATION LTD  
AN 91-203629 [28] WPIDS  
DNC C91-088225

TI Vitamin-E prepn. from vegetable fat and oil - by extracting unsatd.  
oil and fat of vegetable seed, distilling with inert gas,  
heat-distilling in high vacuum and removing solvent, etc..

DC B02 E13

PA (KIKAKU-N) KIKAKU NIICHI KK

CYC 1

PI JP 03127730 A 910530 (9128)\*

ADT JP 03127730 A JP 89-263104 891011

PRAI JP 89-263104 891011

AB JP03127730 A UPAB: 930928

Prepn. of vitamin E: which comprises (1)-(5): is claimed. (1) is  
extn. of unsatd. oil and fat with 70 or more of iodine value or  
dried cpd. of vegetable seed contg. unsatd. oil and fat, using  
solvent; (2) distn. of obtd. extract soln. with addn. of inactive  
gas (e.g. nitrogen); (3) distn. of extract soln. heated in high  
vacuum with stop of gas; (4) final distn. at 200 deg.C. in high  
vacuum of 10 power -6 Torr; and (5) removal of solvent from extract  
soln. to obtain vitamin E.

Pref. the solvent is butanol, ethylene glycol,  
methylethylketone, acetone, benzene or cyclohexane. Fat and oil is  
linseed oil (iodine value: 174-204), sesami oil (195-202), oitica  
oil (140-160), safflower oil (130-150), soybean oil (130-140),  
cotton seed oil (159-170), China or Japanese tung oil (147-173),  
rubber seed oil (128), sunflower oil (125-135), corn seed oil  
(121-130), rape seed oil (94-106), camellia oil (80-82), olive oil  
(80-85), grape seed oil (125-157), tobacco oil, or lemon seed-oil.

USE/ADVANTAGE - Vitamin E from vegetable oil and fat.

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## Tocopherol/Tocotrienol - WPI Results

L22 ANSWER 26 OF 222 WPIDS COPYRIGHT 1997 DERWENT INFORMATION LTD  
AN 89-272229 [38] WPIDS  
DNC C89-120505

TI Prodn. of tocopherols and tocotrienols from palm oil by-prods. -  
Prodn. of tocopherol(s) and tocotrienol(s) from palm oil by-prods..

DC B02 D23 E13

IN KAWADA, T; LEONG, L W; ONG, A S H; TOP, A G; TSUCHIYA, N; WATANABE, H; TOP, A G M

PA (BIDE-N) BIDEC BIOIND DEV CENT; (BIOI-N) BIOINDUSTRY DEV CENTRE; (PALM-N) PALM OIL RES DEV BO; (BIDE-N) BIDEC BIOINDUSTRY DEV CENT; (PALM-N) PALM OIL RES & DEV BOARD

CYC 15

PI EP 333472 A 890920 (8938)\* EN 10 pp

R: AT BE CH DE ES FR GB GR IT LI LU NL SE

AU 8931360 A 890921 (8946)

US 5190618 A 930302 (9311)# 7 pp

ADT EP 333472 A EP 89-302597 890316; US 5190618 A US 89-332238 890331

PRAI AU 88-7273 880316; AU 88-7565 880331; AU 89-31360 890316

AB EP 333472 A UPAB: 930923

Prodn. of tocopherols (T) and tocotrienols (T3) from palm fatty acid distillates (PFAD) is characterised by the following processes: (A) converting the free fatty acids and glycerides in the PFAD into alkyl esters; (B) sepg. T and T3 from the alkyl esters and other impurities; (c) concentrating the T and T3 by ion-exchange; and (D) distilling the prod. to produce a more concd. T and T3 fraction.

ADVANTAGE - The high level of T3 in PFAD is commercially exploited by a combination of unit processes which gives a better yield in better quality than do previous methods.

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## Tocopherol/Tocotrienol - WPI Results

L22 ANSWER 35 OF 222 WPIDS COPYRIGHT 1997 DERWENT INFORMATION LTD  
AN 86-159597 [25] WPIDS  
DNC C86-068475

TI Sepn. procedure of toco tri en ol cpds. - from deodorisation distillate from vegetable oil by methylation and column chromatography.

DC D23 E13

PA (AGEN) AGENCY OF IND SCI & TECHNOLOGY  
CYC 1

PI JP 61093178 A 860512 (8625)\* 3 pp

ADT JP 61093178 A JP 84-214841 841012

PRAI JP 84-214841 841012

AB JP61093178 A UPAB: 930922

Sepn. procedure of tocotrienols from deodorisation and/or deoxidation distillates, obtd. from the purifcn. of vegetable oil(s) and fat(s), comprises methylation of fatty acid(s), and fractionating of tocotrienols by ion-exchange chromatography after or without removal of methyl ester(s) of fatty acid(s).

Deodorisation and/or deoxidation distillate is treated with methanol-sulphuric acid in bubbling nitrogen and fatty acid(s) is methylated. Tocotrienols are sepd. by using ion-exchange chromatography; fatty acid methyl ester(s) may be removed previously or methylated distillate is chromatographed without removal of methyl ester(s). Silica gel chromatography may be combined with ion-exchange chromatography, and solvent extraction may be combined with chromatography.

The vegetable oil is e.g palm oil, rice bran oil, maize oil, etc. Deodorisation and/or deoxidation distillates from these oils contain fatty acid(s), tocotrienols, tocopherols. Methylated distillate is sepd. into tocotrienols and other cpds. by basic anion-exchange chromatography.

The tocotrienol fraction may be purified by silica- and/or alumina -column chromatography, and extraction by lipophilic solvent(s) (e.g. acetone, n-hexane, petroleum ether).

In an example, deodorisation distalliate (1kg), obtaine from rice bran oil, was mixed with methanol (500 ml), 6% sulphuric acid methanol soln. (100 ml) was added dropwise under nitrogen bubbling. The mixture was heated to 60 deg.C under nitrogen. The reaction mixture was washed with water, dried, then fed to an Amberlite CG-400 (RTM) column.

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## Tocopherol/Tocotrienol - WPI Results

L22 ANSWER 38 OF 222 WPIDS COPYRIGHT 1997 DERWENT INFORMATION LTD  
AN 85-273414 [44] WPIDS  
DNC C85-118614

TI High yield tocopherol extraction - from e.g. cotton seed oil bottoms  
by steam distillation under mild conditions.

DC B02 E13

PA (RIKV) RIKEN VITAMIN CO

CYC 1

PI JP 60185776 A 850921 (8544)\* 3 pp

JP 05010349 B 930209 (9309) 3 pp

ADT JP 60185776 A JP 84-41793 840305; JP 05010349 B JP 84-41793 840305

FDT JP 05010349 B Based on JP 60185776

PRAI JP 84-41793 840305

AB JP60185776 A UPAB: 930925

Tocopherol-contg. oil bottoms are subjected to steam-distillation to remove low b.pt.-impurities, so as to make the acid value of the remaining residue 5-20; an alkaline aq. soln. is added to the distilled residue to cause alkaline-purificn.; if necessary, the resultant distilled prod. is further treated with a strong basic anionic exchange resin.

The steam-distillation may be carried out under a moderate conditions (170-230 deg.C, pref. 180-210 deg.C; 30mmHg or less, pref. 20mmHg or less), and so the loss of tocopherol in oil bottoms is very small (3% or less).

In an example, deodorised cotton seed oil bottoms (tocopherol content: 7.3%, acid value: 65.4) (3000g) were subjected to steam distillation at 180 deg.C and 10mmHg, for 2 hrs. to obtain a distilled residue (tocopherol content: 10.3%, acid value: 9.0) (2090g). Percentage recovery 98.3%. 15.0 wt.%-sodium carbonate soln. was added to the distilled residue (2000g) for alkali-purificn., and the sodium soap formed was completely removed by centrifugation and hot water-washing, to obtain an alkali-purified oil (tocopherol content: 10.5%, acid value: 0.4) (1846g). (Percentage recovery 94.1%). This was concd. to obtain a prod. (tocopherol content: 35.6%)  
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## Tocopherol/Tocotrienol - WPI Results

L22 ANSWER 40 OF 222 WPIDS COPYRIGHT 1997 DERWENT INFORMATION LTD  
AN 85-232494 [38] WPIDS  
DNC C85-100741

TI Purificn. of tocopherol - by contacting tocopherol-contg. material with supercritical solvent.

DC B02

PA (MITC) MITSUI PETROCHEM IND CO LTD  
CYC 1

PI JP 60149582 A 850807 (8538)\* 5 pp

ADT JP 60149582 A JP 84-5484 840114

PRAI JP 84-5484 840114

AB JP60149582 A UPAB: 930925

Purificn. of tocopherol comprises contacting a tocopherol-contg. material with a supercritical solvent so that the tocopherol content is enhanced. A feed (e.g. of soybean scum, rice bran, wheat bran, corn oil, malt, internal organs of animals) was fed to the top of a column extractor and the feed is contacted with supercritical solvent. The solvent is liquefied in liquefaction appts, subjected to pressure with pump and fed from a line at the bottom of the column.

Examples of the supercritical solvent are carbon dioxide, ammonia, sulphur dioxide, hydrogen halide, nitrous oxide, hydrogen sulphide, hydrocarbons (e.g. methane, ethane, propane, butane, ethylene, propylene) and halo hydrocarbons (e.g. chloroalkane, chloroalkene, fluoroalkane, fluoroalkene), among which carbon dioxide is most pref.

The supercritical solvent contg. tocopherol is taken from the top of the column and evapd. in a flash-evaporator to collect tocopherol at the bottom of the evaporator. Evapd solvent is recovered.

ADVANTAGE - Method gives tocopherol in high concn. with high purity.

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## Tocopherol/Tocotrienol - WPI Results

L22 ANSWER 42 OF 222 WPIDS COPYRIGHT 1997 DERWENT INFORMATION LTD  
AN 85-102772 [17] WPIDS  
DNC C85-044631

TI High purity tocopherol(s) prodn. - by extraction using supercritical carbon di oxide.

DC B02 E13

PA (SHOW) SHOWA DENKO KK

CYC 1

PI JP 60048981 A 850316 (8517)\* 4 pp

JP 61057310 B 861206 (8701)

ADT JP 60048981 A JP 83-156318 830829

PRAI JP 83-156318 830829

AB JP60048981 A UPAB: 930925

Prodn. comprises extraction of material contg. tocopherols, or a semi-purified prod. with supercritical carbon dioxide. Extraction with carbon dioxide under a pressure higher than its critical pressure 72.8 atmos. (pref. 100 to 300 atmos.) and at a temp. higher than its critical temp. 31 deg. C (pref. 35-75 deg. C) achieves more efficient sepn. of glycerides, terols and other impurities.

USE/ADVANTAGE - Tocopherols can be obtnd. in higher purity (80% or higher) compared with conventional methods (60-70% max.).

In an example, 55.3%-purity crude tocopherols (contg. 40.2% sterols, 2.5% squalene, 1.4% glycerides and other impurities), prepnd. by known methods (solvent extraction and molecular distn.), were supplied through the top of a packed tower 100mm in dia. and 1000mm in packing height at 10Kg/hr., and supercritical carbon dioxide (250 atm., 60 deg. C) was fed through its bottom at 80 Kg/hr to effect countercurrent extraction while maintaining the temp. in the tower at 60 deg. C. The carbon dioxide emerging from the top contained 80.8%-purity tocopherols (yield of 1.2 Kg/hr.). When liq. carbon dioxide of 59 Kg/sq.cm.G and 22 deg. C was used in place of the supercritical carbon dioxide, the purity of tocopherols obtnd. was 76.0% (yield of 80 g/hr.).

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